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(54) Melting/freezing point apparatus

(57) Melting/freezing point apparatus (1) includes means (9) for measuring the change in reflected light from the surface of a substance (7) at the melting point. The apparatus (1) includes a heating block (2) adapted to house a sample plate (4), a light source (8) and a light detector (9). The temperature is measured by a thermocouple in aperture (6). The apparatus may form part of an automated process.

Fig.2.

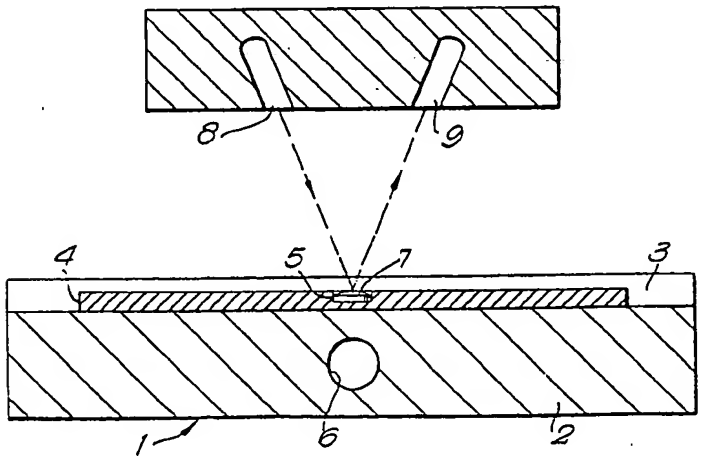

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Fig.1.

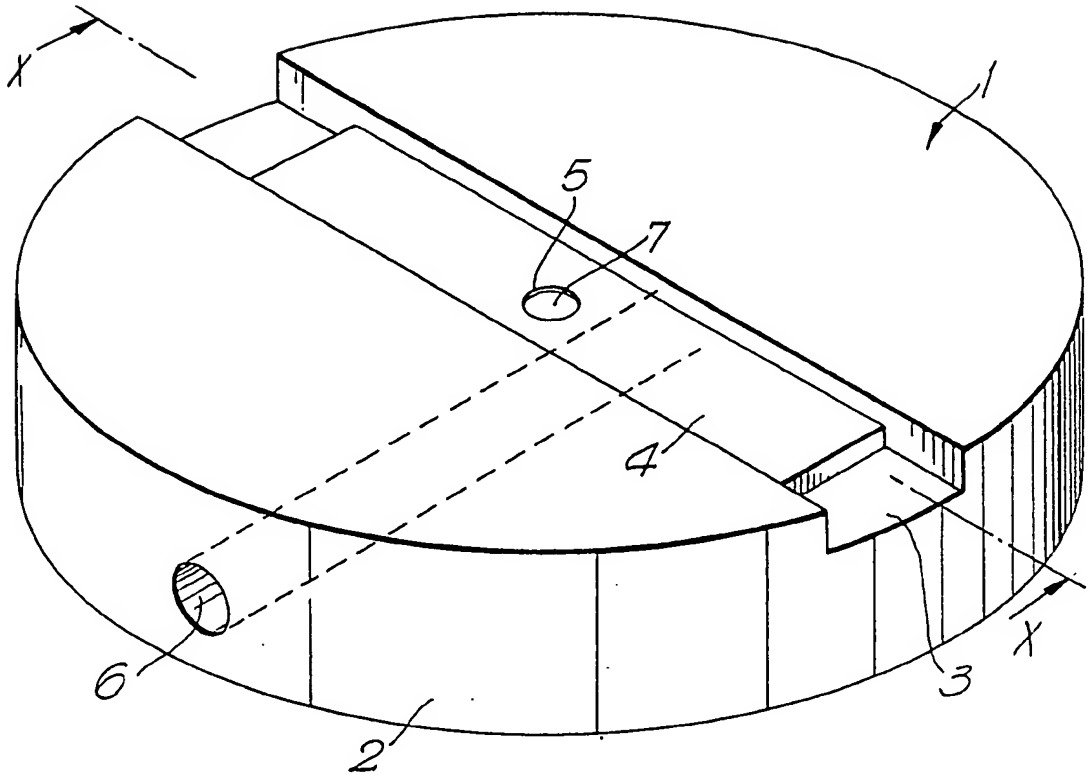
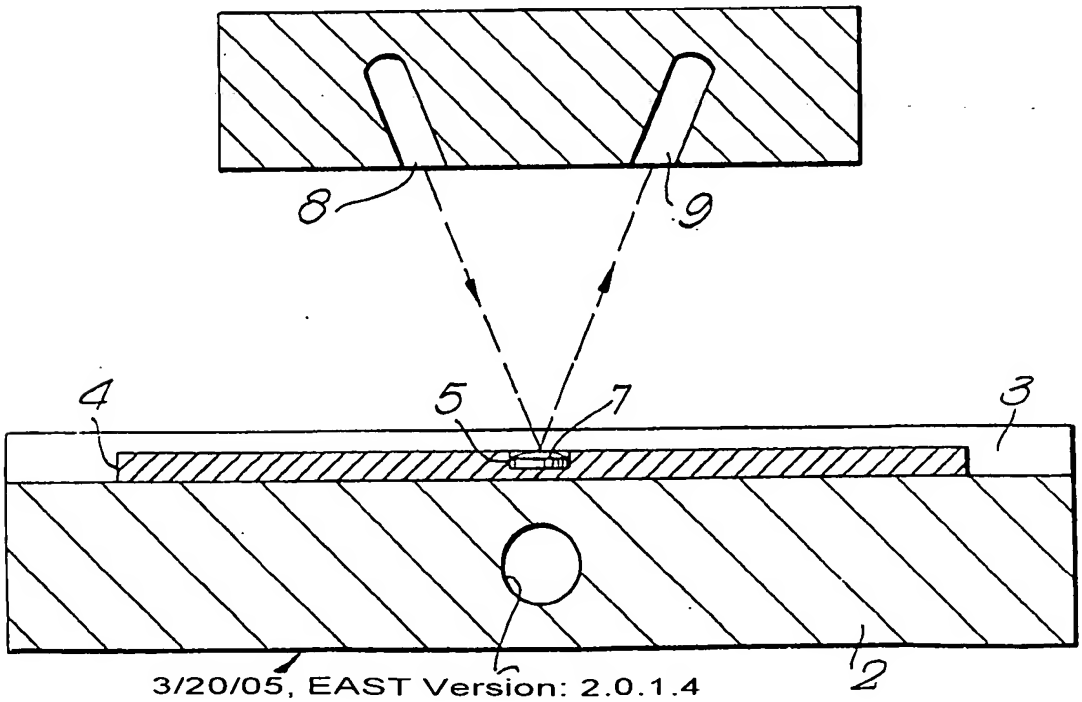


Fig.2.



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Melting Point Apparatus

This invention relates to a new melting point apparatus.

Conventional melting point apparatus require a small solid sample to be placed in a capillary tube and the capillary tube placed in a heated oil bath. This method has the disadvantage that the apparatus must be continually watched in order to determine the point at which the solid sample becomes molten. In addition, with a sample which is damp it is difficult to place sufficient of the sample in the capillary tube. Apparatus have been designed which attempt to overcome these disadvantages. The apparatus described in GB 1246303 measures the change in the optical transmittance of a solid sample upon melting. Similarly, apparatus are known which comprise a heated block wherein the solid sample is placed on a microscope slide. The melting point may be observed through a conventional microscope. However, both of these apparatus require light to be transmitted through the sample. They therefore have the disadvantage that they cannot be used on highly coloured samples or samples which darken at or near the melting point.

Surprisingly, we have now found a means for determining the melting point of a solid which overcomes the disadvantages of existing methods.

According to the invention we provide melting point apparatus which includes means for detecting the change in reflected light from the surface of the substance at the melting point.

The apparatus is particularly suited for the determination of melting point, but may also be used for the determination of the freezing point of a liquid.

The apparatus comprises a sample holder which may be located on or adjacent to a heat source, a light source and a light detector. The light source and light detector are aligned so that a substantial amount of the light reflected from the surface of a substance at its melting point falls on the light detector.

The detector may comprise a simple photoelectric cell but we prefer the detector to be a Schmitt detector. The detector may be linked to a visual and/or audio display unit. In addition the apparatus may be operably linked to a computer such that the rate of heating, the recordal and the display of temperatures and the transference of samples may by automated.

According to the invention we also provide a method of determining the melting point of a substance which comprises detecting the change in reflected light from the surface of the substance at the melting point.

The apparatus of the present invention may be adapted

to measure the melting point of a plurality of samples eg 3, simultaneously. The adapted apparatus may include a plurality of individual sample holders or a single sample holder capable of holding a plurality of samples. The adapted apparatus may comprise a plurality of light sources but the apparatus may include only a single light source. The number of detectors included in the apparatus will depend upon the number of samples being investigated. The apparatus may therefore be used in the analytical technique which measures mixed melting points.

We further provide an automated method of determining the melting point of a substance which comprises use of apparatus adapted to measure the change in reflected light from the surface of the substance at the melting point operably linked to a computer.

When a computer is used to automate the method of melting point determination, we prefer it to control both the rate of heating, the recordal and the display of the transition temperatures and also the transference of samples.

Any conventional heat source may be used or a light source may by used, eg an infra-red light, to provide heat. We prefer the heat source to comprise a thermally conductive block.

When the apparatus is utilised in freezing point

determination any conventional cooling means may be used but we prefer to use a thermally conductive block which may be cooled, eg at one end.

The maximum melting point of a solid substance which may be recorded will depend upon the heat source used but the apparatus may be useful for measuring melting points up to 600°C, preferably 500°C and more preferably 450°C.

The minimum freezing point which may be determined will depend upon the coolant used but the apparatus may be useful for measuring freezing points down to -200°C, preferably -78°C, more preferably -30°C and most preferably 0°C.

The melting point may be measured by conventional means, eg by mercury or alcohol thermometer, or preferably by a thermocouple.

The light source may emit light of a number of wavelengths, eg white light, or of a specific wavelength. The light source may also act as a heat source, eg by emitting infra-red light.

The invention will now be illustrated by way of example only and with reference to the accompanying drawings in which :

Figure 1 is a perspective view of a heating block, and

Figure 2 is a sectional view of a heating block taken

along the line X-X including a sectional view of the light source and detector.

Referring to Figures 1 and 2, the melting point apparatus (1) comprises a cylindrical heating block (2) provided with a diametric groove (3) adapted to house a rectangular sample plate (4). The sample plate (4) comprises a strip of thermally conductive material provided with a well (5) capable of holding a solid sample (7) of which the melting point is to be determined. The heating block (2) is also provided with a radial aperture (6) at right angles to the diametric groove (3), adapted to house a temperature detector eg a thermocouple, beneath the well (5) of plate (4) during operation.

For the determination of melting point, a light source (8) and a light detector (9) are located adjacent to the heating block (2) and arranged such that light falling on the solid sample (7) is scattered and little or no response is observed by the detector (9). When the sample plate (4) reaches the melting point of the solid sample (7), the sample (7) becomes molten. The change from solid to molten form alters the characteristic of the sample (7) towards light. In particular the surface of the molten sample (7) is reflective towards light. Thus, an increase in the intensity of light is observed at the detector (9). The temperature at this point can be

determined by the temperature detector, eg a thermocouple inserted in the aperture (6).

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What we claim is:-

1. Melting point apparatus which includes means for detecting the change in reflected light from the surface of a substance at the melting point.

2. Apparatus according to Claim 1 in which the light detector is a Schmitt detector.

3. Apparatus according to any one of the preceding claims, in which the detector is linked to a visual and/or audio display unit.

4. Apparatus according to Claim 3, wherein the apparatus is linked to a computer and the determination of melting point is automated.

5. A method of determining the melting point of a substance which comprises detecting the change in reflected light from the surface of a substance at the melting point.

6. An automated method of determining the melting point of a substance, which comprises use of apparatus adapted to measure the change in reflected light from the surface of the substance at the melting point operably linked to a computer.

7. A melting point apparatus comprising a cylindrical heating block provided with a diametric groove adapted to house a rectangular sample plate, the sample plate comprising a strip of thermally conductive material

provided with a well capable of holding a solid sample of which the melting point is to be determined, the heating block is also provided with a radial aperture, at right angles to the diametric groove, adapted to house a temperature detector beneath the well of the sample plate during operation, a light source and a light detector are located adjacent to the heating block.

8. An apparatus substantially as described herein with reference to the accompanying drawings.

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